

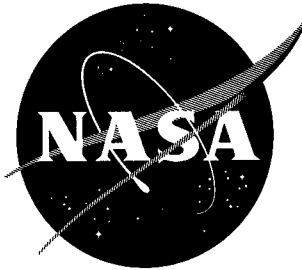
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# TECHNICAL NOTE

## D-1646

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EFFECTS OF SPUTTERING WITH HYDROGEN IONS ON TOTAL  
HEMISPHERICAL EMITTANCE OF SEVERAL  
METALLIC SURFACES

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SUMMARY

Experiments were conducted to measure the change in <sup>all</sup> total hemispherical emittance of several metallic specimens due to sputtering with hydrogen ions. The test specimens were cylindrical and were constructed from pure titanium, a titanium alloy containing 6-percent aluminum and 4-percent vanadium, pure aluminum, 2024 aluminum alloy, and pure copper. The energy level of the incident ions was 1000 electron volts. The test specimens were subjected to ion bombardment by immersion in a hydrogen plasma. The total hemispherical emittance of each specimen was measured in a cold-wall, vacuum-type calorimeter. *end work*

Five specimens were electropolished and one specimen was sandblasted before ion bombardment. Emittance was measured both before ion bombardment was initiated and after successive bombardment periods until a total of  $10^{21}$  ions/cm<sup>2</sup> had bombarded each specimen. For the five initially polished specimens, the test results indicated that, for the total test period, the emittance of the pure aluminum increased the most (about 60 percent from its initial value), whereas, the emittance of both the pure and alloyed titanium remained essentially unchanged. During the total test period, the emittance of the initially sandblasted copper specimen decreased to one-third of the unspattered value.

Photomicrographs of all test specimens before and after bombardment with  $10^{21}$  ions/cm<sup>2</sup> are presented and discussed. In addition, the changes in weight resulting from ion bombardment of all specimens are presented.

The time required to achieve significant changes in emittance for surfaces in the spatial environment and in an ion engine is discussed briefly. *end*

INTRODUCTION

The rate at which heat can be radiated from a surface is directly proportional to the total hemispherical emittance of that surface (see ref. 1). Thus, any changes in the emittance of a spacecraft's surface due to the spatial

environment will cause corresponding changes in heat radiated from that surface and in surface temperatures. (Definitions of the radiative heat-transfer terms are given in ref. 2 and those terms used in this report are defined in appendix A.)

The environmental factors which influence metals applications in space vehicles are discussed in reference 3. This reference indicates that the spatial environment contains a large number of high-energy particles which can bombard a space vehicle. This bombardment can cause the ejection of surface atoms and this result is commonly called sputtering. A considerable amount of work has been done on sputtering in relation to the number of surface atoms ejected per incident ion for various kinds of bombarding ions, ion energies, and surface materials. (See, e.g., ref. 4.) However, nothing has been found in the literature to indicate the effects of sputtering on the emittance characteristics of a surface.

The emittance of a surface is strongly dependent on the finish of that surface as noted in references 5 and 6 and, thus, could be altered by sputtering. Therefore, an experimental program was initiated to determine the effects of ion bombardment on the total hemispherical emittance of several metallic surfaces. Since the majority of ionized particles in the solar system is protons (see ref. 3), ionized hydrogen was chosen as the bombarding ion. This report presents the results of this experimental program.

## EQUIPMENT

The equipment for the test program consisted of a sputtering apparatus, an emittance-measuring apparatus, and certain auxiliary equipment. A description of each apparatus follows.

### Sputtering Apparatus

The principle of cathode sputtering (see ref. 7) was employed in this apparatus. For this application a target was immersed (similar to a large negative Langmuir probe) in a hydrogen plasma. The kinetic energy of the ions was controlled by the magnitude of the negative voltage applied to the target with respect to the plasma. A grid around the target provided the reference potential and also shielded the target from the ion sheath.

A cutaway view of this apparatus is shown in figure 1. A vacuum system with a 4-inch-diameter oil diffusion pump maintained the desired test pressure in the 5-inch bell jar. The cylindrical specimen was located in the center of the bell jar and was the negative electrode or target. The specimen mount had the necessary piping and seals to allow the circulation of cooling air at room temperature and pressure on the interior surface of the specimen. A stainless-steel grid (mesh size 16) surrounded the specimen and was grounded to the base plate. Hydrogen gas was bled into the bell jar and was ionized by a single-side-band, radio-frequency transmitter. This transmitter was operated in a continuous wave (cw) mode and supplied power to a resonant circuit at 28 Mcps. The ion accelerating potential was supplied by an external direct-current power supply set at

1000 volts. The ion current was measured with a recording ammeter. This type of sputtering apparatus is capable of producing an ion current of about  $1 \text{ ma/cm}^2$ .

### Emittance-Measuring Apparatus

The emittance-measuring apparatus employed the calorimetric technique whereby the total hemispherical emittance of a body was determined from the steady-state radiative heat flow from that body to another completely enclosing body. Figure 2 is a cutaway view of this apparatus.

The entire cold-wall enclosure and specimen assembly were contained within a standard 18-inch bell jar. The chamber was evacuated by a vacuum system which utilized a 4-inch-diameter oil diffusion pump, a mechanical pump, and liquid-nitrogen-cooled baffles. The cold-wall enclosure was a 0.062-inch-thick stainless-steel shell about 10 inches in diameter and 10 inches in length. The interior surface of the enclosure was first sandblasted and then sprayed with three coats of black sicon paint. Each coat was baked for two hours at  $100^\circ \text{C}$  before the next coat was applied. A  $3/8$ -inch-diameter, stainless-steel, liquid-nitrogen cooling coil was soldered to the outside of the enclosure.

The test specimen was heated with an internal electrical heater element (see fig. 3). This element had 18-ohms of 22-gage nichrome wire wound around a boron nitride core. Two lead wires of number 26 copper wire 6 inches long were connected at one end to the nichrome element and at the other end to number 14-gage electrical lead wires from a d-c power supply. The current and voltage measurements required to evaluate the power input were made with the aid of a potentiometer and standard resistors.

The specimen was suspended within the cold wall by the number 26-gage lead wires as shown in figure 2. To minimize the heat conduction losses along the specimen power leads, the leads were clamped in a guard heater at the point where they passed through the cold-wall enclosure, as indicated. This heater was a chromium plated copper body with a heater element of 5-ohms of 22-gage nichrome wire wound on a boron nitride core. The assembly of the specimen and its guard heater is shown in figure 4.

### Auxiliary Equipment

The auxiliary equipment consisted of polishing equipment, sandblasting equipment, an ion accelerator, and a metallograph. The electropolishing equipment consisted of a d-c power supply, timer, beaker, cylindrical cathode, cooling bath, and solution agitator. The cathode, which was placed concentrically around the specimen for electropolishing, was constructed from perforated stainless steel and was about 2 inches in diameter and 4 inches in length. The sandblasting equipment was a commercial unit designed to blast-clean a surface with very fine abrasives. The 8 kv mass-analyzed ion accelerator described in reference 4 was used to determine the hydrogen ion species present in the bell jar of the sputtering apparatus. For this test, the ion jug of the mass-analyzed ion accelerator

was replaced with the bell jar and R-F excitation equipment from the sputtering apparatus. Visual inspection and photographs of the specimen surface were made with the aid of a metallograph. Tungsten or zirconium arc lamps were used to illuminate the specimen and the angle of illumination was held constant so that comparisons of photomicrographs could be made.

## SPECIMEN GEOMETRY, MATERIALS, AND PREPARATION

### Geometry

The specimen size and configuration were dependent on various requirements. It was necessary for the specimen to be able to accommodate an internal heater or cooler, to have sufficient surface area so that accurate emittance measurements could be made, and to be free from sharp corners or edges so that all ion bombardment would be normal to the surface. In addition, the entire surface must be uniformly sputtered for an accurate evaluation of emittance. It was decided that a hollow cylindrical specimen was compatible with these requirements. Figure 5 shows the geometry of this specimen with pertinent dimensions.

### Materials

Five materials were selected for test: 99.9-percent pure titanium, titanium alloy containing 6-percent aluminum and 4-percent vanadium, 99.99-percent pure aluminum, aluminum alloy (2024), and electrolytic copper. The alloys of aluminum and titanium were selected as representative structural materials. The pure aluminum and titanium were selected to indicate the possible differences in emittance obtained on a pure and alloyed material. The copper was chosen since it has been used in previous sputtering tests (refs. 4 and 8).

### Surface Preparation

Two types of initial surface finish were used in the tests: One was an electropolished surface, the other a sandblasted surface. Each process was divided into two parts: the prefinish and the final finish.

The amount of surface roughness which could be removed by the electropolishing process in a reasonable time was determined by microscopic examination. If no visible traces of the initial surface roughness remained after electropolishing, the prefinish was considered to be adequate. Abrasive papers and felt cloths with various grits were used for the prefinish. The final grit depended upon the hardness of the specimen material. Table I lists the finest grit used for each material as well as the original form of the material and the final finish.

The development of a proper electropolishing technique for each material required much experimenting to determine the proper voltage, current density, time

of run, bath composition and temperature. Table II gives the various parameters used for the five materials. In addition, the amount of agitation of the solution influenced the final finish.

To eliminate the uncertainties which might have resulted from using two different specimens, the sandblasted specimen was the same specimen tested with the initially polished surface. The surface was first sanded with abrasive paper so that it would be a clean and uniform surface (see table I), free from all effects of previous tests. The surface was then blasted with glass beads of mesh size MS-L270 with an air pressure of 95 psi. The gun nozzle was held approximately 5 inches from the specimen surface during the blasting operation. A sweeping motion similar to that in spray painting was used to cover the surface. The specimen was rotated between each pass.

### TEST PROCEDURE

The procedures for sputtering the specimens and for determining their emittances were essentially the same for all materials. First, the emittance was determined for the initially prepared surface. Photomicrographs were taken and the specimen was weighed. Then, the specimen was subjected to several periods of sputtering. The specimen was weighed, photomicrographs were made, and the total hemispherical emittance was measured after each sputtering period. This procedure was arbitrarily continued until about  $10^{21}$  ions/cm<sup>2</sup> had bombarded each specimen.

### Sputtering Procedure

After a specimen had been installed in the sputtering apparatus, the apparatus was purged of all contaminants. First, the bell jar was evacuated to a pressure of  $1 \times 10^{-5}$  mm Hg and checked for leakage. Then the hydrogen supply line was flushed and, with the evacuating system running, hydrogen was bled into the system at a rate which maintained the pressure in the bell jar at 0.05 mm Hg for a period of 5 minutes. The hydrogen flow rate was then reduced to the operating pressure of 0.01 mm Hg.

Sputtering was initiated by ionizing the gas with R-F excitation and by applying a negative voltage potential to the specimen. The transmitter was then tuned to obtain the maximum ion current. To maintain the specimen temperature below 250° F, air was circulated through the interior of the model while it was being sputtered.

The sputtering period between sequential emittance measurements was arbitrarily established as that time required for the specimen to collect a charge of 2000 coulombs. With the average current of 40 milliamperes for these tests, each sputtering period was about 15 hours. Since this apparatus was not automatically controlled, it was necessary to build up the sputtering period from three runs with a maximum time of 6 hours per run. For storage between runs during any period, the apparatus was normally just evacuated to, and maintained at,  $1 \times 10^{-6}$  mm Hg. For the copper specimens, the build up of sputtered material on the bell

jar during a sputtering period was sufficient to interfere with the inductive coupling of the R-F field with the hydrogen gas. In these cases, it was necessary to clean the bell jar at least once during a sputtering period. At the completion of a sputtering period, the specimen was cooled to room temperature and then dry nitrogen was bled into the system to raise the pressure to atmospheric pressure. The specimen was then removed, weighed and photographed and, where possible, immediately installed in the emittance-measuring apparatus. Otherwise, it was stored at room temperature in a dessicating cabinet.

### Emittance-Measuring Procedure

Total hemispherical emittance measurements were obtained in the following manner: First, a 0.005-inch iron-constantan thermocouple was either spot welded or swaged on the surface of the specimen. The guard heater and specimen heater were then installed and the assembly was placed inside the cold-wall enclosure. Next, the entire system was evacuated to approximately  $1 \times 10^{-5}$  mm Hg and liquid nitrogen coolant was admitted into the cold-wall coils. Finally, power was applied to the specimen and guard heaters and was adjusted until the specimen equilibrium temperature was  $100^{\circ} \text{C} \pm 10^{\circ} \text{C}$  and the guard heater temperature was within  $\pm 15^{\circ} \text{C}$  of the specimen temperature. The current and voltage necessary to maintain the specimen temperature were then recorded. These measurements were used to calculate the power input to the specimen and, in turn, to determine the specimen emittance.

### DATA REDUCTION AND ACCURACY

#### Sputtering

Two parameters most commonly used in discussions of sputtering data are the number of incident ions,  $N_i$ , and the weight change,  $\Delta M$ , of a specimen during a sputtering period. From these data, it is possible to calculate a third parameter called sputtering yield,  $Y$ , which is the ratio of the number of atoms sputtered from a surface to the number of incident ions,  $N_a/N_i$ .

Number of incident ions.— The number of incident ions can be computed from the time of test and the total current that flows between the specimen and grid. The total current has two components, (a) the current due to impingement by the incident ions and (b) a secondary current resulting from the emission of electrons from the specimen surfaces due to the impinging ions. Thus, the number of incident ions per unit surface area may be computed from the equation

$$N_i = \left( \frac{I}{1 + \gamma} \right) \frac{t}{eA_s} \quad (1)$$

where  $\gamma$  is the electron secondary emission coefficient. (A complete list of symbols is presented in appendix B.)

For a typical sputtering period the total current and time of sputtering could be recorded within an accuracy of 1 percent. The data in references 9 and 10 indicate that the secondary emission coefficient for hydrogen ion bombardment is probably less than 15 percent. Since no reasonable correction could be made for the secondary current in these tests,  $\gamma$  is assumed to be zero. Thus, the number of bombarding ions (computed from eq. (1)) may be in error by as much as 15 percent.

Weight change.- The weight change due to ion bombardment was determined by weighing the specimen before and after a sputtering period on a semimicrobalance with a sensitivity of 0.005 milligram. To evaluate the actual experimental error in weight change, a copper specimen was subjected to the previously described procedure for sputtering a specimen but the accelerating potential was never applied. Thus, the weight change of the specimen due to exposure to the atmosphere, handling, installation and removal from the apparatus, contamination by cooling air, etc., was measured. The resulting maximum variation in specimen weight was  $\pm 0.06$  mg; for a normal 2000 coulomb sputtering period the error of  $\pm 0.06$  mg amounts to only about 0.2-percent error in weight change of copper which had the highest weight change of the materials tested. For pure aluminum, which had the lowest weight change, the corresponding error is about 13 percent.

To minimize the effects of variable temperature on specimen weight change, the specimen equilibrium temperature was maintained in the range from 80° to 250° F by circulating air on the interior surface of the specimen. Data in reference 11 indicate that temperature variations in this range would cause a negligible error in weight change.

Weight change per unit surface area,  $\Delta M$ , is computed by dividing the measured weight change by the surface area of the specimen,  $A_s$ .

Sputtering yield.- Sputtering yield is calculated from the equation

$$Y = \frac{N_a}{N_i} = \left( \frac{\Delta M}{M_a} \right) \left( \frac{A_{se}}{t} \right) \left( \frac{1 + \gamma}{I} \right) \quad (2)$$

It can be seen from the above discussions on number of incident ions and on mass loss that sputtering yields for the present tests can be in error by as much as 35 percent. Since the main purpose of the test program was to determine the effects of hydrogen ion bombardment on total hemispherical emittance, no refinements in test apparatus or procedure were made to determine more exact sputtering yields.

#### Emittance Measurements

The net power diffusely radiated from a completely convex body (specimen) to a completely concave enclosure at a lower temperature (cold wall enclosure) may be calculated from Christiansen's equation (ref. 12)



$$q_{\text{net}} = \frac{1}{1 + \epsilon_s \left( \frac{1}{\epsilon_c} - 1 \right) \frac{A_s}{A_c}} \epsilon_s \sigma A_s (T_s^4 - T_c^4) \quad (3)$$

For the present experiment, the specimen area ( $A_s$ ) was 40.05 cm<sup>2</sup>, and the area of the enclosure ( $A_c$ ) was approximately 3030 cm<sup>2</sup> or about 75 times as large. The emittance of the enclosure ( $\epsilon_c$ ) was 0.90 ± 0.05 as measured on a test sample. Therefore, since the emittance of the specimen ( $\epsilon_s$ ) cannot be greater than 1.0, the quantity  $\epsilon_s[(1/\epsilon_c) - 1](A_s/A_c)$  is less than 0.0024 and equation (3) may be simplified to

$$q_{\text{net}} = \epsilon_s \sigma A_s (T_s^4 - T_c^4) \quad (4)$$

or

$$\epsilon_s = \frac{q_{\text{net}}}{\sigma A_s (T_s^4 - T_c^4)} \quad (5)$$

Since the specimen surface temperature was measured by a thermocouple, a test was made to determine its proper location so that its reading would be representative of the entire surface. If the emittance of the specimen is assumed to be uniform, temperature gradients are dependent upon the design of the heater element and the thermal conductivity of the material. A material with low thermal conductivity, stainless steel, was selected to evaluate the possible gradient. Three thermocouples were mounted on the specimen, one at the center and one near each end. The specimen was then mounted in the test apparatus and a typical run conducted. In all cases the indicated temperatures of all three thermocouples were within ±1° C; it was, therefore, concluded that the placement of the thermocouple was not critical for these tests.

The total amount of power supplied to the lead wires of the apparatus included not only the power radiated from the specimen,  $q_{\text{net}}$ , but also all power losses (ohmic, thermal conduction, and radiation) from these power leads. To calculate  $q_{\text{net}}$ , corrections for these losses were made and the possible errors in these corrections were included in the error analysis.

The errors involved in making emittance measurements of this kind may be divided into two groups, systematic and random. A systematic error analysis, similar to the one in reference 13, was performed on the entire apparatus and readout equipment; the results indicated  $\Delta\epsilon/\epsilon$  was less than 0.02 for the emittance range of the materials tested. The principal source of error was the uncertainty associated with the absolute magnitude of the specimen surface temperature. After several emittance measurements of the same specimen surface had been made, the random errors in some cases were discovered to be greater than the limits calculated from the predictable systematic errors. Based upon a careful study of the test results, it was estimated that the maximum uncertainty in absolute emittance was ±0.002 for all materials with emittances of 0.1 or less. For materials with an emittance greater than 0.1, the maximum uncertainty was within the limit set by the above systematic error analysis. It should be emphasized here that this figure represents the accuracy with which the emittance of a specific surface may be measured and is not indicative of the accuracy of surface reproduction.

## RESULTS AND DISCUSSION

The results of this experimental program are presented as follows: First, discussion and evaluation tests of the sputtering apparatus; second, the emittance change of the six test specimens as a function of number of bombarding hydrogen ions; third, weight change; and, finally, the effect of various environments on ion bombardment time.

### Discussion and Evaluation of Sputtering Apparatus

Two common types of sputtering apparatus are the mass-analyzed ion beam (e.g., ref. 4) and the glow-discharge-type apparatus (e.g., ref. 7). The mass-analyzed ion beam apparatus described in reference 4 was considered for the proposed tests but the low current density, the small beam diameter, the specimen shape, and the nonuniformity of the sputtered area made this apparatus impractical. The glow-discharge-type sputtering apparatus was selected because it had several features that were particularly useful in the proposed experiment. It, of course, also had certain limitations.

The sputtering apparatus selected was capable of producing an ion current density of about 1 ma/cm<sup>2</sup> which was sufficient to produce measurable surface effects in a reasonable length of time. The corresponding bombardment rate was also large compared to the bombardment rate of the residual gases in the chamber and, therefore, the effects of bombardment by the residual gases were minimized. Another very important feature of this apparatus was its ability to uniformly sputter the surface of a large specimen.

One disadvantage of a glow-discharge-type apparatus is the possible back-scattering of sputtered atoms to the specimen surface if the discharge is operated at excessively high pressures. To establish the operating pressure for this apparatus, a test was conducted to determine the effect of pressure on yield. It was found that at operating pressures below 0.015 mm Hg, the yield was independent of pressure and, thereby, indicated negligible back-scatter at lower pressures. During the operation of the equipment at the selected operating pressure of 0.010 mm Hg, it was also noted that material sputtered from the specimen formed a sharp image of the grid on the bell jar, which indicated a straight line path of the sputtered material from the specimen to the bell jar. This was additional verification of negligible back-scatter.

Further disadvantages of a glow-discharge apparatus are that secondary electron emission from the specimen is difficult to suppress, the bombarding energy is difficult to control, and charge exchange and neutral particle bombardment may occur. In this experiment the suppression of secondary electrons from the specimen was not feasible; however, errors associated with this effect have been discussed in the section on data reduction and accuracy. A test was conducted to evaluate the possible errors due to energy control, charge exchange, and neutral particle bombardment. Data on sputtering yield obtained with this apparatus and similar results obtained with a mass-analyzed ion accelerator (see ref. 4) are presented in figure 6. Since no appropriate sputtering yield data associated

with hydrogen ion bombardment were available, these tests consisted of bombarding copper specimens with argon ions at various energy levels in the 150 to 1600 electron volt range. It can be seen that the sputtering yields were essentially the same at each ion energy level for the two types of ion accelerators. Since the ion beam is generally considered a more precise instrument and since our results favorably agree with those obtained with this instrument, it was concluded that the total effect of the errors associated with this glow-discharge-type sputtering apparatus was negligible and that the apparatus shown in figure 1 was a satisfactory design for these tests.

Since the glow-discharge-type sputtering apparatus does not separate various species of ions, another test was performed to mass analyze the hydrogen gas plasma generated in the sputtering apparatus. For this test, the bell jar, grid, and R-F coil (with the associated R-F power supply) were mounted on the ion accelerator described in reference 4. The results indicated that the plasma contained 5 to 15 percent  $H^+$ , 5 to 20 percent  $H_2^+$ , and 60 to 90 percent  $H_3^+$ . These percentages depended on a number of parameters, such as R-F field strength, amount of sputtered material deposited on the inside of the bell jar, gas pressure, etc. Small amounts of  $N^+$ ,  $O_2^+$ , and other ions were detected; however, their concentration was several orders of magnitude less than that of the hydrogen ions. For the operating conditions of the sputtering apparatus, these tests indicated that the majority of bombarding ions was  $H_3^+$  and that some changes in plasma composition probably occurred within a sputtering period.

The yield produced by one  $H_3^+$  ion may be assumed to be approximately the same as that produced by three  $H^+$  ions with the same total energy; that is, each  $H^+$  ion would have one-third the energy of the  $H_3^+$  ion. Similarly, an  $H_2^+$  ion will sputter approximately the same amount of material as two  $H^+$  ions with the same total energy. Experimental results from reference 4 for  $N^+$  and  $N_2^+$  on copper and reference 14 for  $H^+$ ,  $H_2^+$ ,  $H_3^+$  on silver show the assumption to be valid within 20 percent. The rate of surface erosion (yield) may then be approximated for a specific hydrogen ion species by employing this assumption.

In a similar manner, it was assumed for the tests in this report that the emittance change of a material bombarded by a specific hydrogen ion may be approximated if the rate of change of emittance is assumed to be independent of the ion energy (i.e., for the ion energy range of 330 ev for  $H^+$  to 1000 ev for  $H_3^+$ ). This has not been experimentally verified; however, for a first approximation it appears to be a reasonable assumption.

#### Emittance Change

In order to determine the change in total hemispherical emittance of several metallic surfaces when bombarded by hydrogen ions, it was necessary that the species of bombarding ions and the ion energy be maintained as constant as possible for all sputtering periods. The gas pressure and R-F field strength were constant for all tests and the ion energy was maintained constant at 1000 electron volts. This value was chosen because a survey of the literature indicated that 1000 electron volts is near the sputtering maximum for hydrogen. The results of this experimental program are presented in figures 7 through 12.

The total hemispherical emittance of the pure titanium and the titanium alloy as a function of number of bombarding ions is presented in figure 7 and photomicrographs of both surfaces before and after bombardment are shown in figure 8. It can be seen that bombardment by  $10^{21}$  ions/cm<sup>2</sup> caused essentially no change in either emittance or surface condition of either material. However, as will be discussed later, the weight changes of the specimens indicated that some surface material was removed by sputtering.

The emittance of the pure aluminum and the aluminum alloy as a function of number of bombarding ions is shown in figure 9. For the pure aluminum, the emittance increased to a value about 60 percent greater than the emittance of the polished surface and then remained essentially constant with increased sputtering. For the aluminum alloy, the emittance decreased during the first sputtering period. The reason for the decrease is not known but is believed to be due to the sputtering away of a thin surface film left from the electropolishing process. After the first sputtering period, the emittance of the aluminum alloy increased with increasing numbers of bombarding ions and, when the test was terminated at about  $10^{21}$  ions/cm<sup>2</sup>, the emittance had not as yet approached a constant value. The maximum change in emittance of the aluminum alloy recorded during the test period was about 50 percent.

Figure 10 shows photomicrographs of the aluminum surfaces before and after ion bombardment. In the pure aluminum, the crystal boundaries were defined and many small pits were formed by the sputtering process. The photomicrographs of the aluminum alloy before and after sputtering show the presence of the alloying elements. These elements appear to have been sputtered away at a faster rate than the base material and to have formed cavities in the surface. Note again that the sputtering has defined the crystal boundaries. For both the pure and alloyed aluminum, the sputtering rate on all the crystals appears to be quite uniform.

The emittance of two pure copper specimens as a function of number of bombarding ions is presented in figure 11. One specimen was initially electropolished; the other was initially sandblasted and had an emittance about 10 times greater than that of the electropolished specimen. The data on the initially electropolished specimen indicate that the emittance increased by 35 percent as a result of sputtering. However, due to the very low emittance value of polished copper, the actual percentage increase is anywhere from 13 to 58 percent because of the inaccuracy of the measurement. (See section on data reduction and accuracy.) After the first sputtering period, the emittance remained essentially constant throughout the rest of the test. In contrast, the emittance of the sandblasted specimen was decreased to one third of its initial value by the bombarding action of about  $10^{21}$  ions/cm<sup>2</sup>. It can be seen in this figure that the emittance of the polished specimen and the emittance of the sandblasted specimen appear to approach a common value as the number of bombarding ions is increased. This convergence should be expected since enough material should eventually be removed to make the surface characteristics independent of initial surface finish.

Photomicrographs of both copper specimens before and after ion bombardment are shown in figure 12. It will be noted in figure 12(a) that the surfaces of different crystals of the initially polished specimen were preferentially sputtered. The surface of each crystal is well defined and there are relatively large variations in the height between adjacent crystal surfaces. This

preferential sputtering has been discussed in reference 8 and is believed to result from surface atoms being ejected easier in some directions than in others and is dependent upon the crystal structure and the orientation of this structure with respect to the surface of the test specimen. The surface finish produced by the sandblasting process on a copper specimen is shown in the top photomicrograph of figure 12(b). The lower photomicrograph shows the same surface after bombardment with  $10^{21}$  ions/cm<sup>2</sup>. In this case, the coarse texture of the specimen before bombardment was reduced to a finer texture after bombardment. It can be seen from figures 11 and 12 that although the emittance is approaching a common asymptote with increasing numbers of bombarding ions, the surface textures are not as yet the same. The sandblasting process probably changed the surface properties and only after all of this altered portion of the surface has been removed can the surface texture resemble that of figure 12(a) after ion bombardment.

### Weight Change

The weight change per unit surface area for each of the six test specimens as a function of number of bombarding ions is shown in figure 13. In general, the copper specimens experienced the greatest weight change and the aluminum specimens the smallest. Also, all of the specimens except the pure titanium experienced a mass loss as a result of the sputtering process. The weight of the pure titanium specimen actually increased with increasing number of bombarding ions.

The reasons for the contrasting weight changes of the pure titanium and the titanium alloy are not known. In most sputtering tests, the weight change of a specimen is believed to be due principally to the ejection of surface atoms (see refs. 4 and 15). However, results of the present tests on pure titanium indicated that the hydrogen ions may have been occluded in the lattice structure or adsorbed on the surface (a phenomenon which is characteristic of these two elements as indicated in ref. 16) and that the rate of ejection of titanium atoms per incident ion was low. Thus, the weight of the pure titanium sample increased apparently because the weight gain due to hydrogen occlusion and adsorption was greater than the weight loss due to sputtering. In the case of the titanium alloy, the weight loss due to sputtering was apparently greater than the weight gain due to hydrogen occlusion and adsorption.

The differences in weight change per incident ion of the two copper specimens shown in figure 13 are believed to result from the differences in surface finish. During the initial sputtering period the mass loss per incident ion was greater for the polished specimen than for the sandblasted specimen. It is believed that this difference is a result of the surface properties produced by the sandblasting process. During the later sputtering periods, the mass loss per incident ion for the initially polished copper surface was almost constant; whereas the corresponding mass loss for the sandblasted specimen is still increasing. Eventually, the mass loss per incident ion should be the same for the two specimens, that is, when all the effects of the initial surface finish have been removed.

It has been shown that weight change due to ion bombardment can vary over wide ranges, depending on both the base material and surface finish. When one

considers the emittance data (figs. 7, 9, and 11) and the weight change data (fig. 13) it can be seen that, although the aluminum specimens had the smallest weight change, the corresponding emittance changes were the greatest. Thus, it is apparent that the weight change of a material due to sputtering should not be used as a criteria for predictions of the change in emittance of that material.

#### Effect of Various Environments on Ion Bombardment Time

The length of time required for a surface to be bombarded by a specific number of ions depends, of course, upon the ion flux density of its environment. Current estimates from reference 17 indicate that the spatial environment during a quiet sun contains about  $10^9$  protons/cm<sup>2</sup> sec and the average energy of the particles is about 1.3 kev. During solar maxima, the flux may increase by three orders of magnitude. If a surface is subjected to this spatial environment during solar maxima ( $10^{12}$  protons/cm<sup>2</sup> sec), the full scale of the abscissa ( $1 \times 10^{21}$  ions/cm<sup>2</sup>) in figures 7, 9, 11, and 13 corresponds to a time of about 30 years. If it is assumed that the integrated effect of the solar wind (protons plus other heavier ions) is equivalent to that of the hydrogen plasma in this experiment, it appears that, for the aluminum and copper surfaces, times of about a year could cause significant emittance changes.

If the environment has the much higher flux which might be encountered in an ion propulsion system, the time required to bombard a surface with  $1 \times 10^{21}$  ions/cm<sup>2</sup> could be reduced to hours. Thus, each material with its particular surface finish must be evaluated in its particular environment to determine whether the emittance and, hence, the thermal energy balance of that surface will be significantly altered.

#### CONCLUDING REMARKS

It has been shown that hydrogen ion bombardment can significantly change the total hemispherical emittance of metallic surfaces.

Test results from copper surfaces indicate that when ion bombardment of a surface is initiated, the emittance can either increase or decrease, depending on the initial surface finish (e.g., polished or sandblasted). It also appears that as the ion bombardment continues the emittance of a sputtered surface approaches a constant value which is independent of the initial surface finish.

Weight changes of specimens bombarded with hydrogen ions can vary over wide ranges, depending on the base material and surface finish. Test results indicate that the weight change of a material due to sputtering should not be used as a criteria for predictions of the change in emittance of that material.

According to current estimates of the hydrogen ion flux in the spatial environment, times on the order of a year or more may be required to appreciably change the emittance of a metallic skin on a space vehicle. However, other

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environments, such as those in ion propulsion engines, may subject a surface to considerably higher fluxes and the corresponding times may be reduced to hours.

Ames Research Center  
National Aeronautics and Space Administration  
Moffett Field, Calif., Oct. 4, 1962

## APPENDIX A

### RADIATIVE HEAT-TRANSFER TERMS

The definitions of radiative heat-transfer terms used in this report were taken from reference 2.

Radiant flux is the rate of flow of radiant energy. It is analogous to current as applied to electricity.

Emittance is a property of a specimen; it is the ratio of its rate of emission of radiant energy to that of a black-body radiator at the same temperature under the same conditions.

Emissivity is a special case of emittance; it is a fundamental property of a material and is measured as the emittance of a specimen of the material that has an optically smooth surface and is sufficiently thick to be opaque.

Hemispherical is referred to as emission from a surface in all possible directions.

The word total, as used to modify terms describing thermal emission characteristics, means that the modified terms pertain to the integrals of rates of spectral emission at all wavelengths. An example of a qualified expression that is required to convey a precise description is "total hemispherical emittance."



## APPENDIX B

### SYMBOLS

A	area, cm <sup>2</sup>
e	electronic charge, $1.6 \times 10^{-19}$ coulomb
I	total measured current, amps
M <sub>a</sub>	weight of one atom of sputtered material, grams
$\Delta M$	weight change per unit surface area, $\frac{\text{weight after sputtering minus weight before sputtering}}{A_s}$ , grams/cm <sup>2</sup>
N <sub>a</sub>	number of sputtered atoms per unit area
N <sub>i</sub>	number of incident ions per unit area
q <sub>net</sub>	net power radiated from enclosed body, watts
t	time, sec
T	temperature, °K
Y	yield, atoms/ion
$\gamma$	electron secondary emission coefficient
$\epsilon$	total hemispherical emittance
$\sigma$	Stefan-Boltzman constant, $5.6686 \times 10^{-12}$ watts/cm <sup>2</sup> deg <sup>4</sup>

### Subscripts

c	cold-wall enclosure
s	specimen surface

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TABLE I.- SPECIMEN MATERIAL, PREFINISH AND FINAL FINISH

Specimen number	Material	Original form	Prefinish	Final finish
1	Pure titanium	Rod	600 grit aluminum oxide powder on felt	Electro-polish
2	Ti-6Al-4V	Bar	600 grit silicon carbide paper	Electro-polish
3	Pure aluminum	Rod	600 grit aluminum oxide powder on felt	Electro-polish
4	Aluminum 2024	Rod	600 grit aluminum oxide powder on felt	Electro-polish
5	Pure copper	Rod	Crocus cloth	Electro-polish
6	Pure copper	Rod	600 grit silicon carbide paper	Sandblast

TABLE II.- PARAMETERS FOR ELECTROPOLISHING TEST SPECIMENS

Material	Voltage, volts	Current, amps	Time, sec	Temp., °F	Solution	Reference for solution
Pure copper	10	10	50	60-80	Phosphoric acid 250 cc Ethanol 250 cc Propanol 50 cc Water 500 cc Urea 3 g	a
Pure titanium	40	30	30	35	Methanol 490 cc Ethylene glycol 440 cc Perchloric acid* 45 cc Water 30 cc	b
Titanium alloy Ti-6Al-4V	25	20	50	40	Methanol 590 cc Butyl Cellusolve 350 cc Perchloric acid* 60 cc	a
Pure aluminum	30	25	40	35	Perchloric acid* 62 cc Ethanol 700 cc Butyl Cellusolve 100 cc Water 137 cc	a
Aluminum alloy 2024	30	25	40	40	Perchloric acid* 62 cc Ethanol 700 cc Butyl Cellusolve 100 cc Water 137 cc	a

\*Perchloric acid (70 percent). Specific gravity 1.67.

a Electropolishing Manual, Buehler Ltd., Evanston, Illinois.

b Gurklis, J. A., McGraw, L. D., Faust, C. L.: Electropolishing and Chemical Polishing of High-Strength, High-Temperature Metals and Alloys. DMIC Memorandum 98, April 1961.

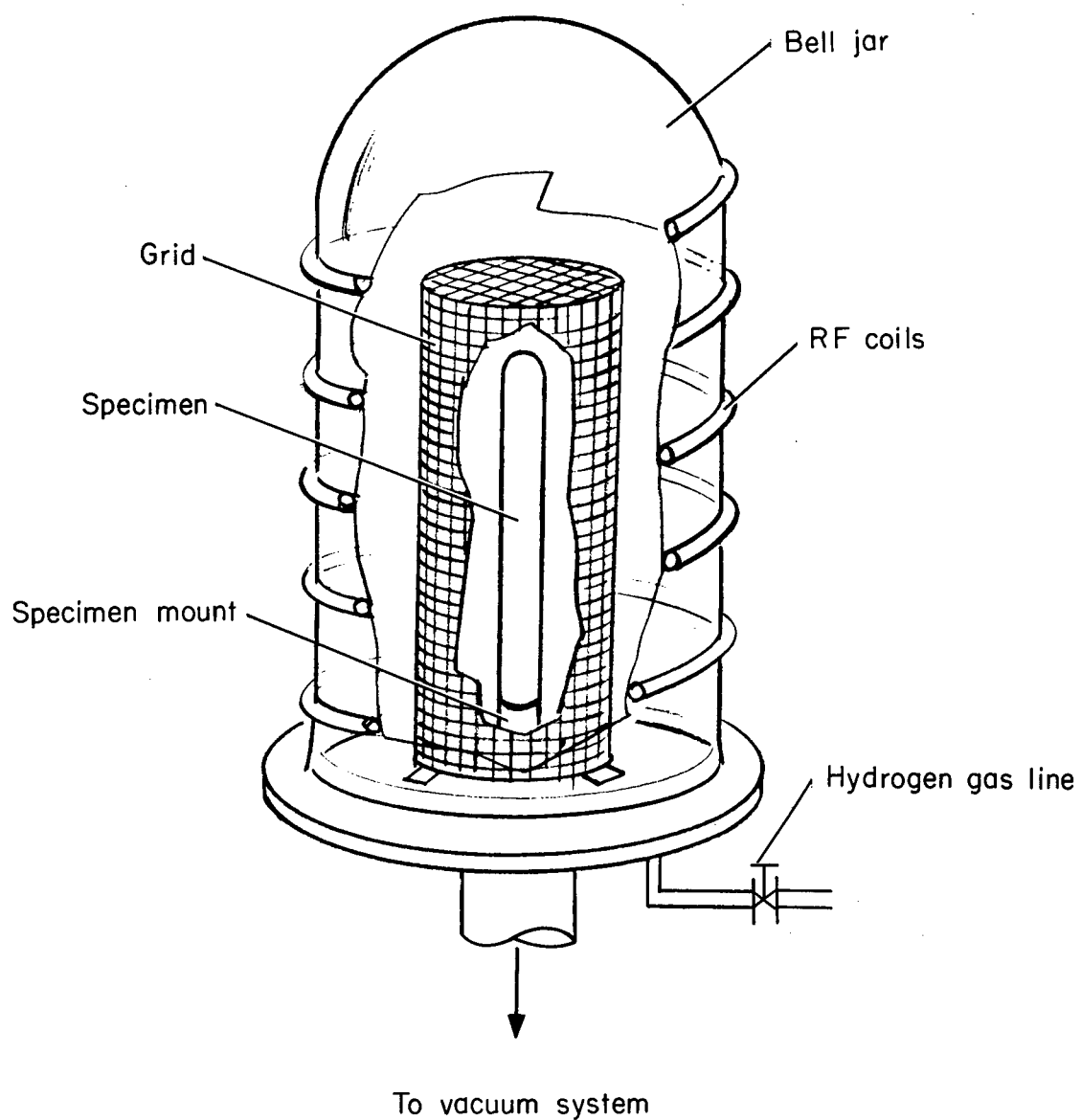


Figure 1.- Cutaway view of sputtering apparatus.

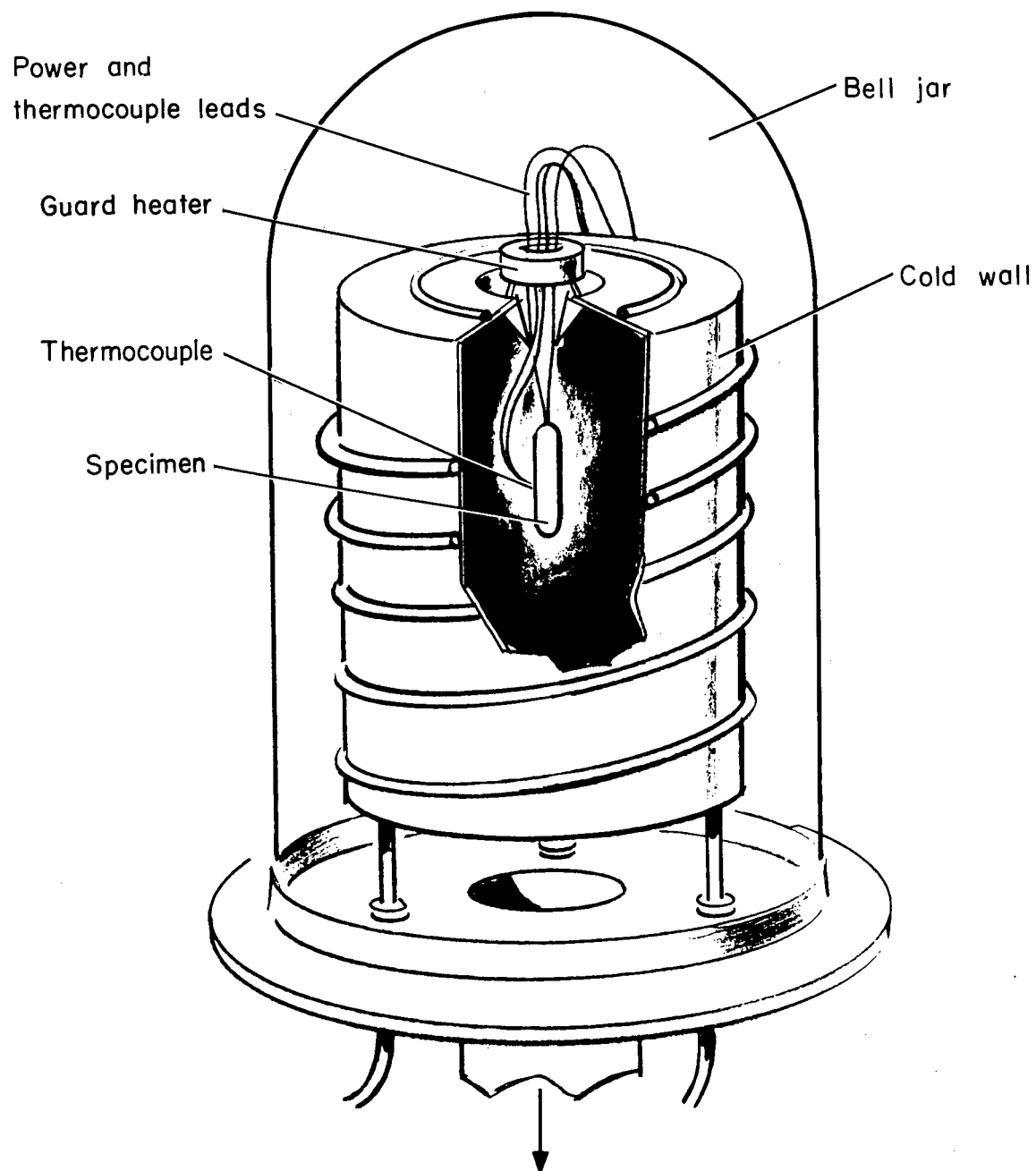


Figure 2.- Cutaway view of total hemispherical emittance measuring apparatus.

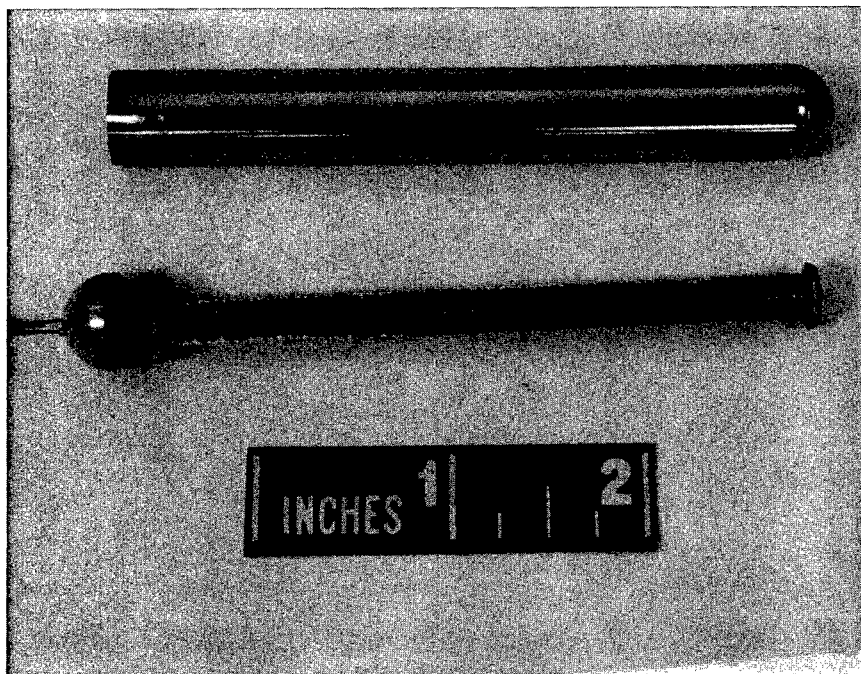


Figure 3.- Photograph of typical specimen and specimen heater.



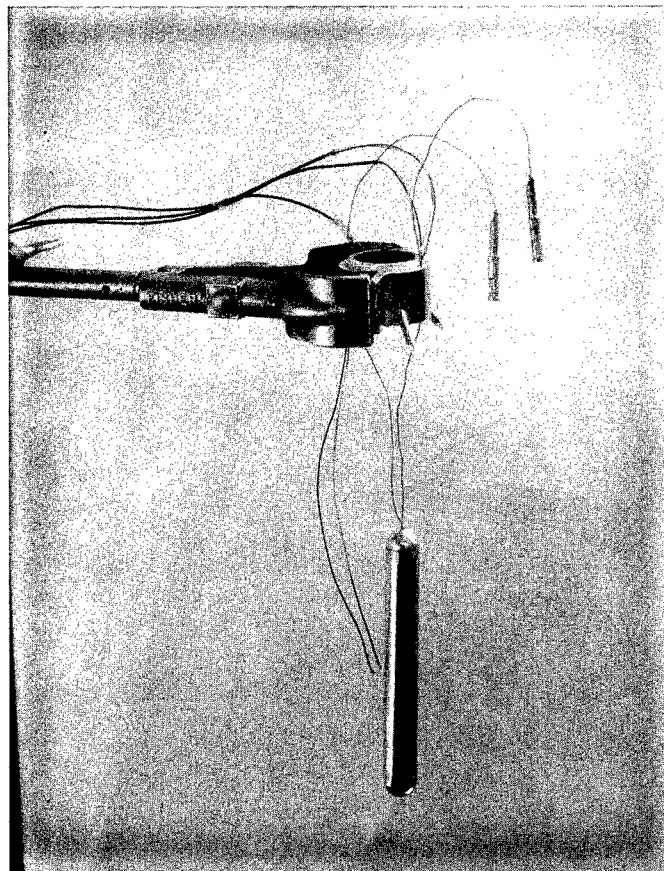
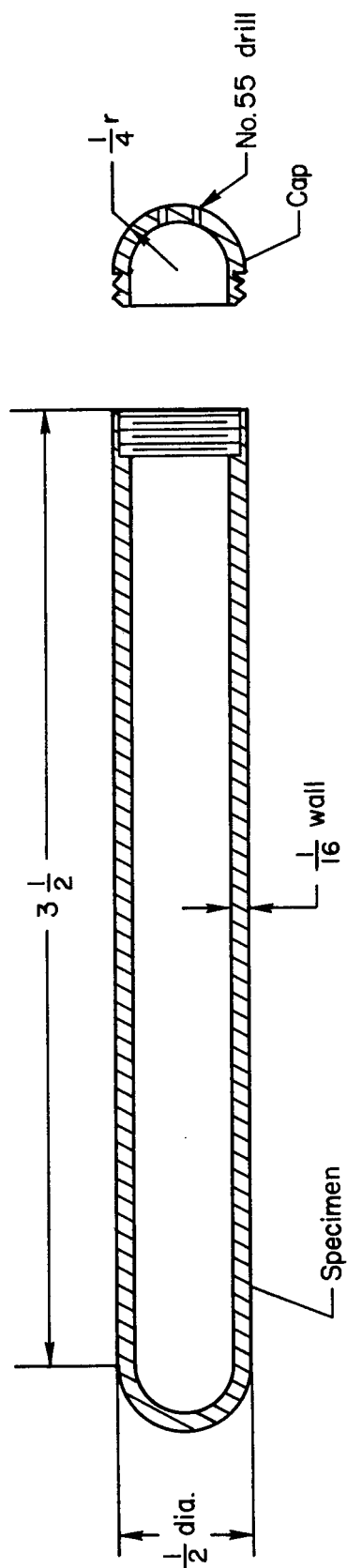


Figure 4.- Photograph of typical specimen and guard heater assembly.



Surface area 6.28 in.<sup>2</sup>

Note: All dimensions in inches.

Figure 5.- Geometry and dimensions of test specimen.

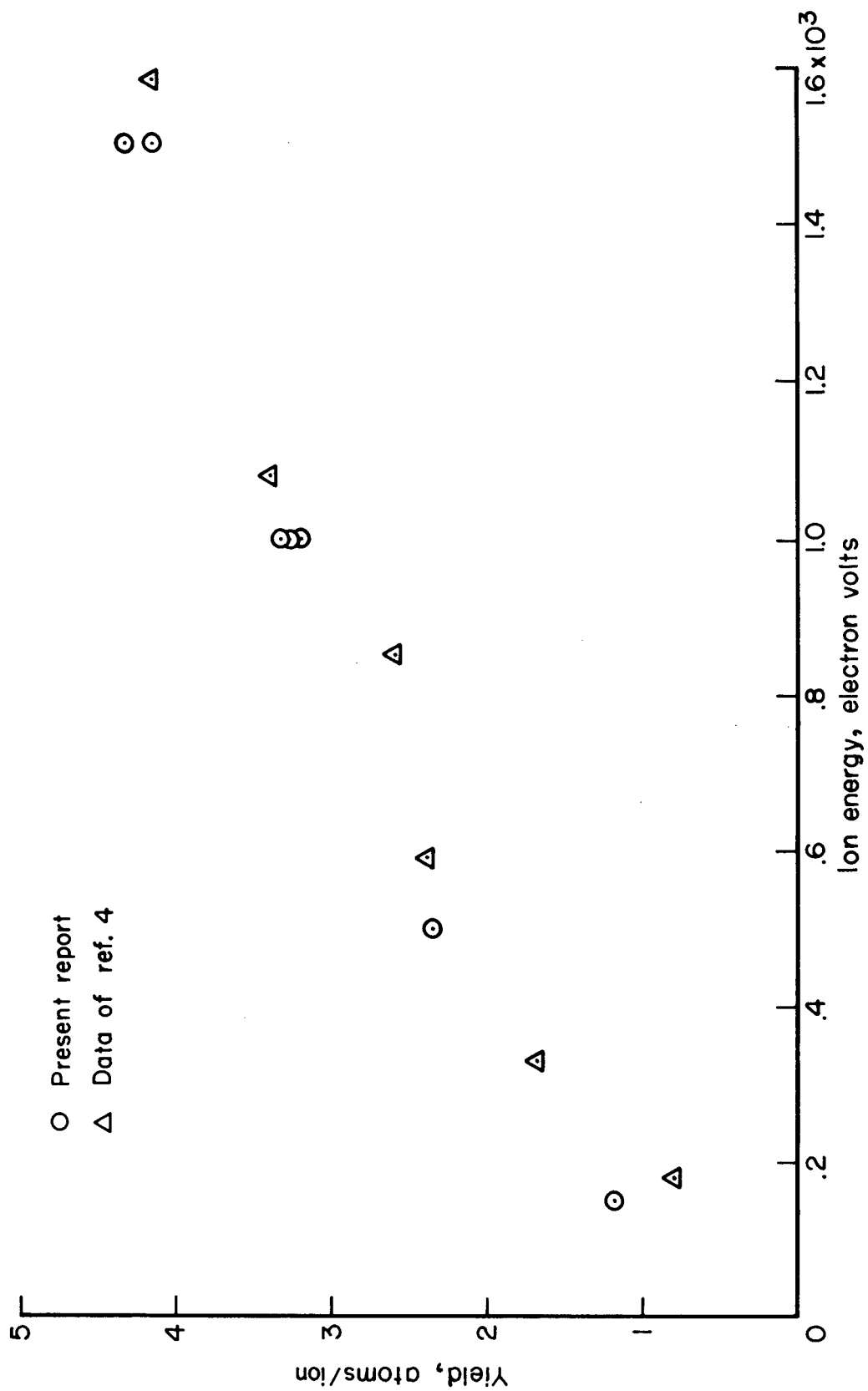


Figure 6.- Sputtering yield of copper bombarded by argon ions as a function of ion energy (normal incidence).

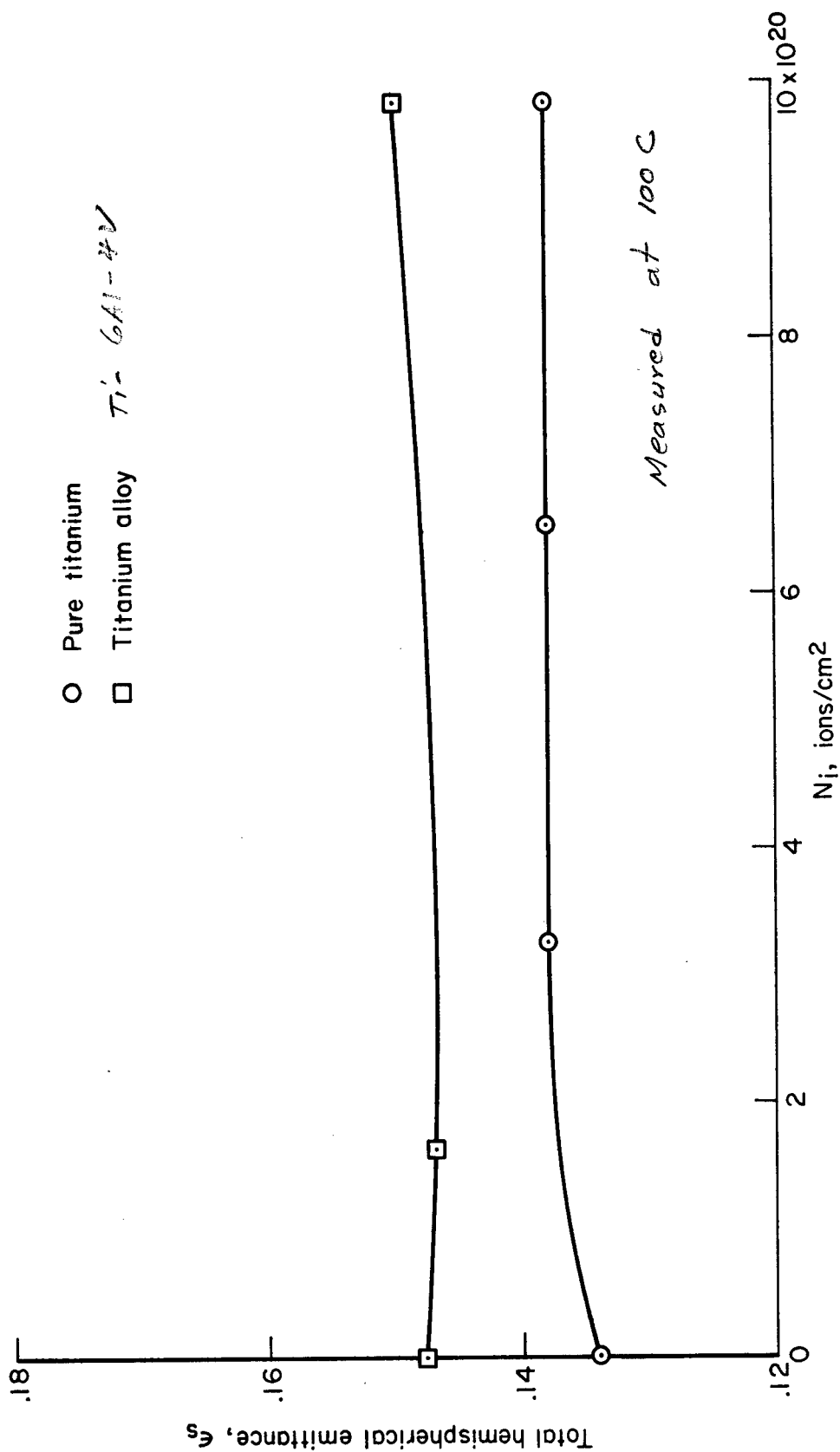
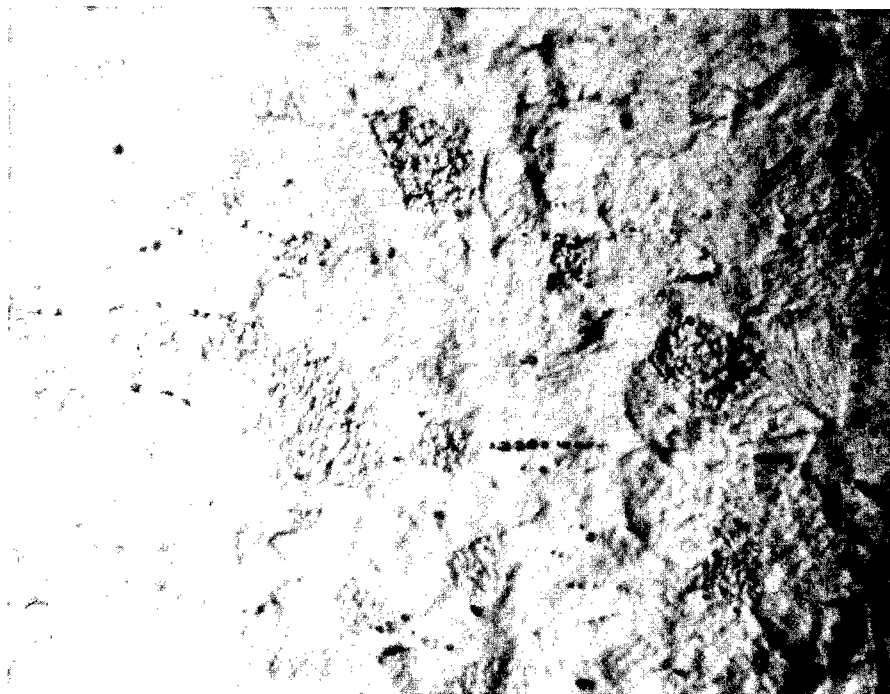


Figure 7.- Effect of hydrogen ion bombardment on total hemispherical emittance of titanium.



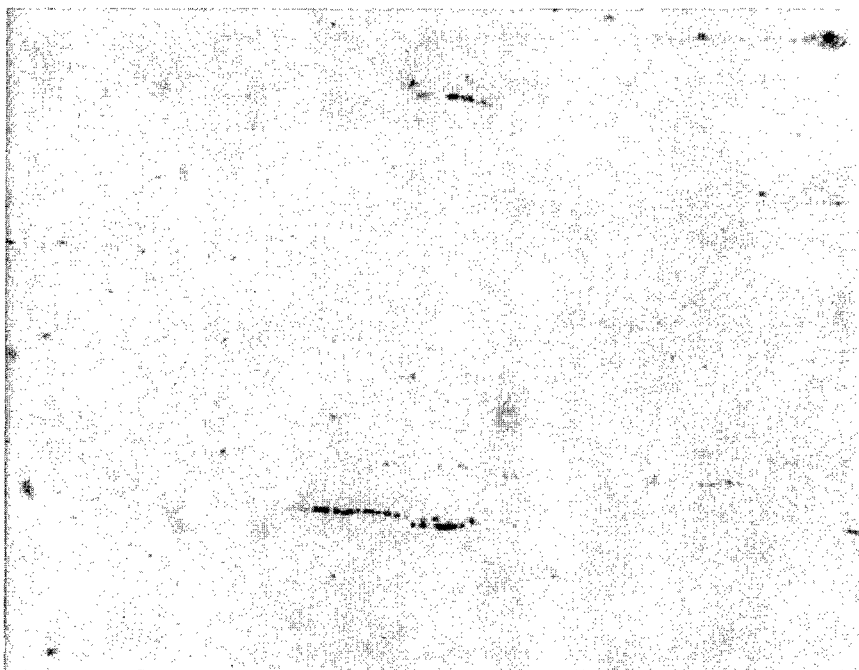
Before ion bombardment.



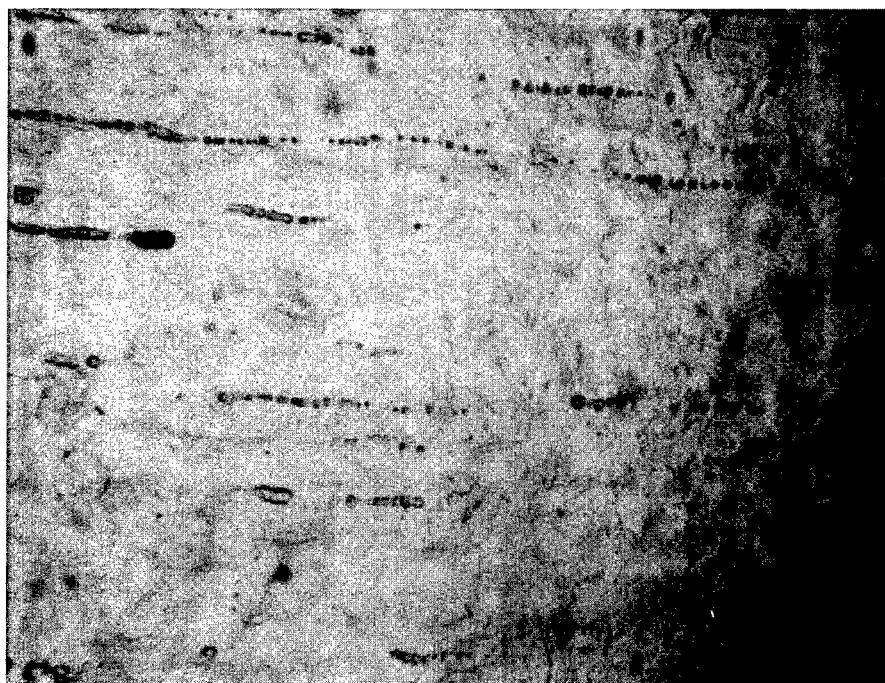
After bombardment with approximately  $10^{21}$  ions/cm<sup>2</sup>.

(a) Pure titanium.

Figure 8.- Photomicrographs of titanium specimens before and after sputtering (500 X).



Before ion bombardment.



After bombardment with approximately  $10^{21}$  ions/cm<sup>2</sup>.

(b) Titanium alloy, Ti - 6Al - 4V.

Figure 8.- Concluded.

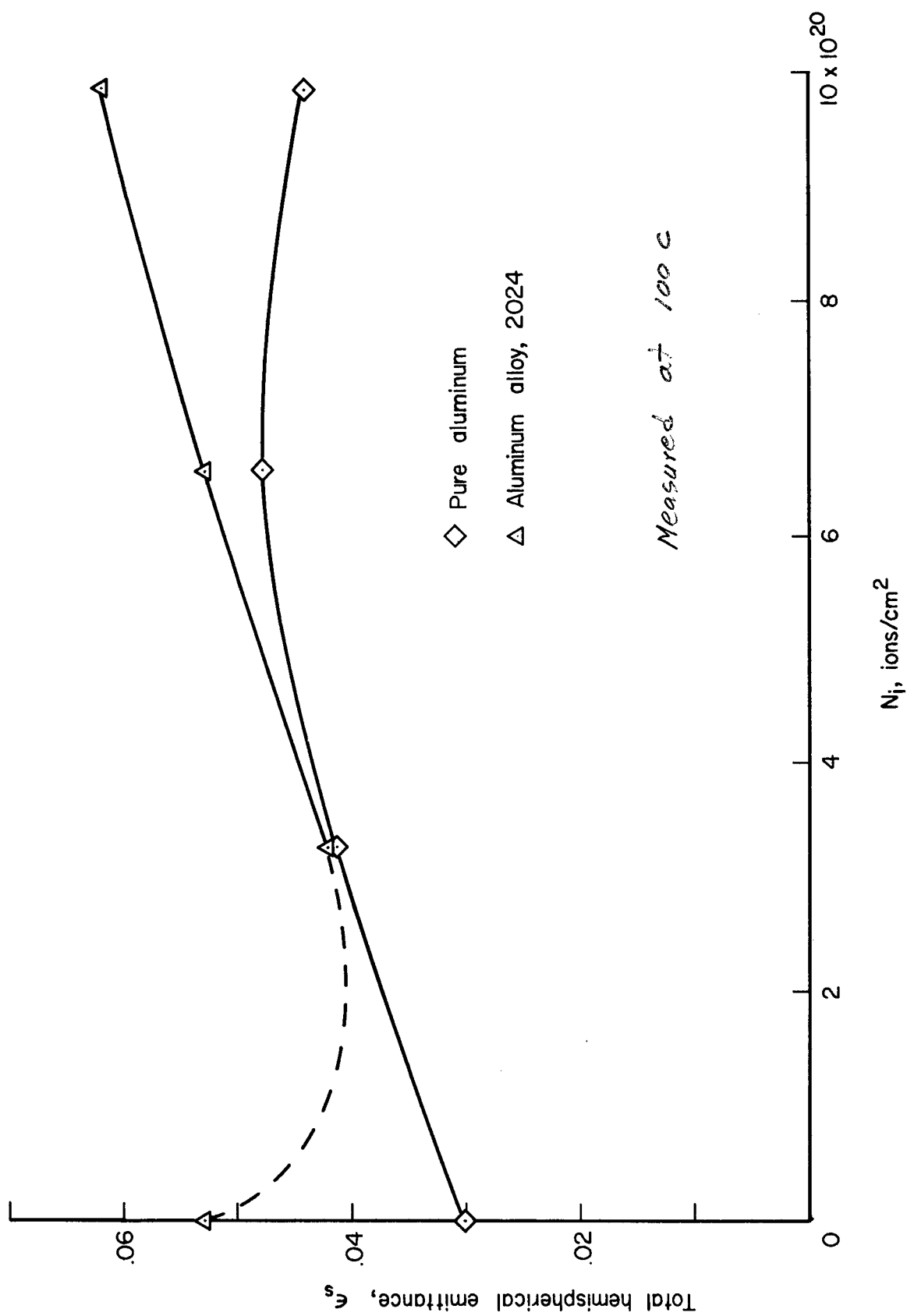
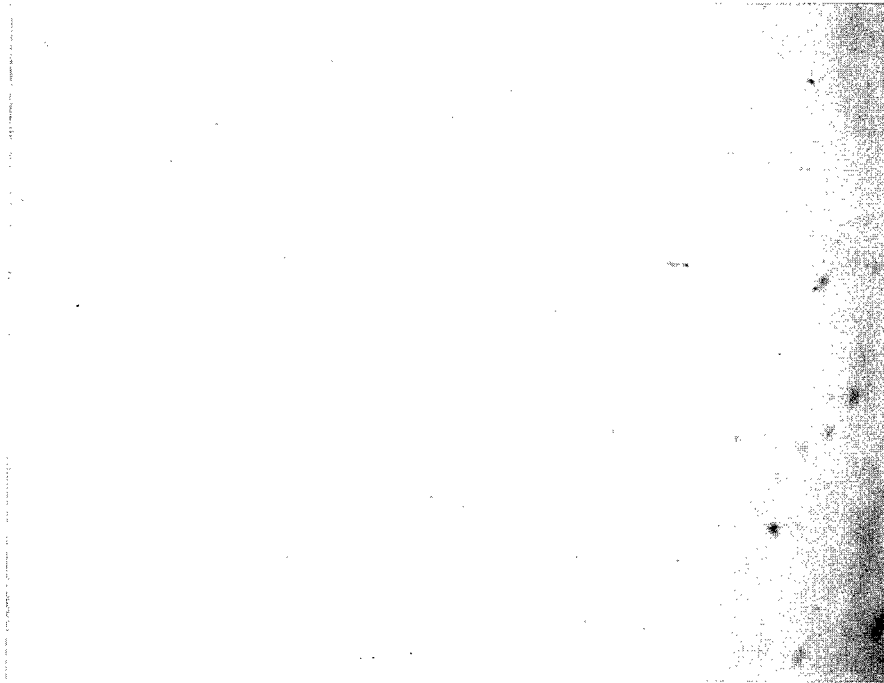
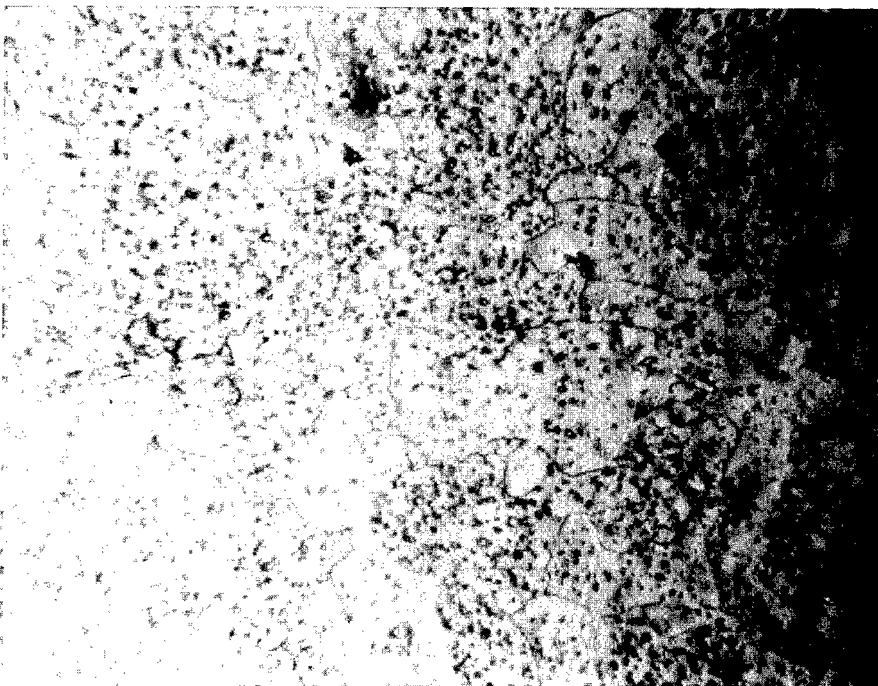


Figure 9.- Effect of hydrogen ion bombardment on total hemispherical emittance of aluminum.



Before ion bombardment.

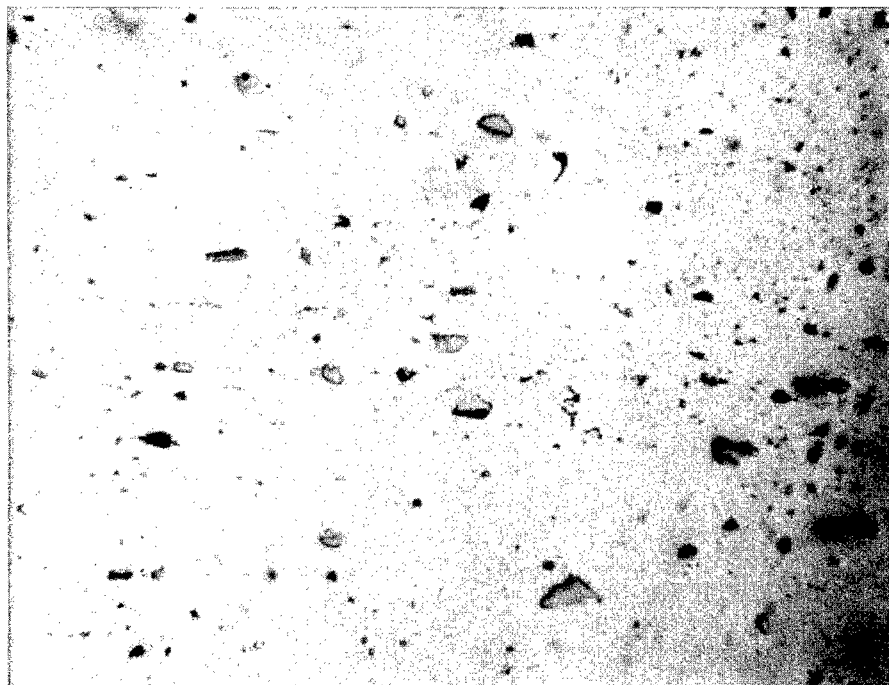


After bombardment with approximately  $10^{21}$  ions/cm<sup>2</sup>.

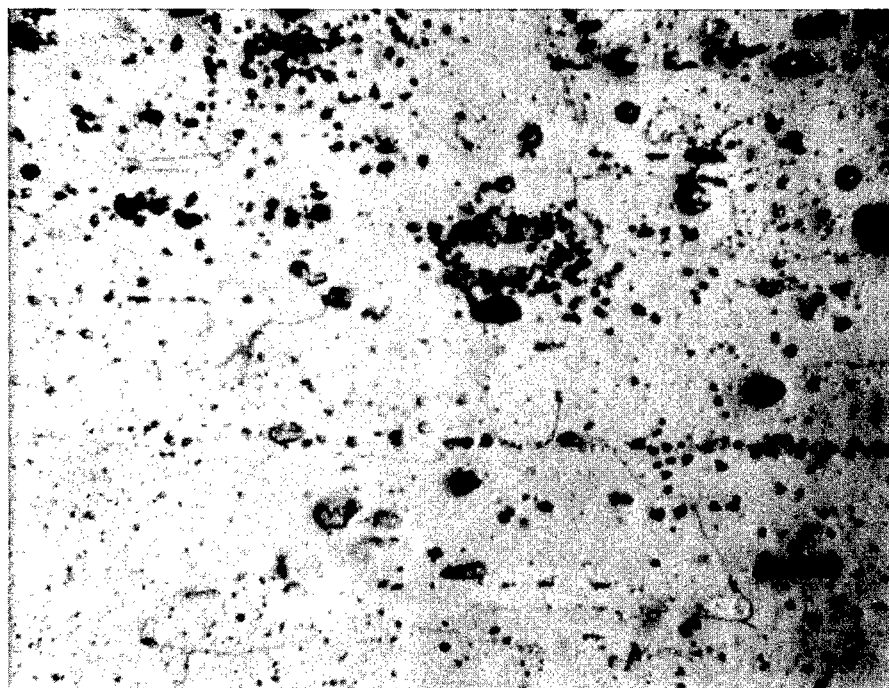
(a) Pure aluminum.

Figure 10.- Photomicrographs of aluminum specimens before and after sputtering (500 X).





Before ion bombardment.



After bombardment with approximately  $10^{21}$  ions/cm<sup>2</sup>.

(b) Aluminum alloy, 2024.

Figure 10.- Concluded.

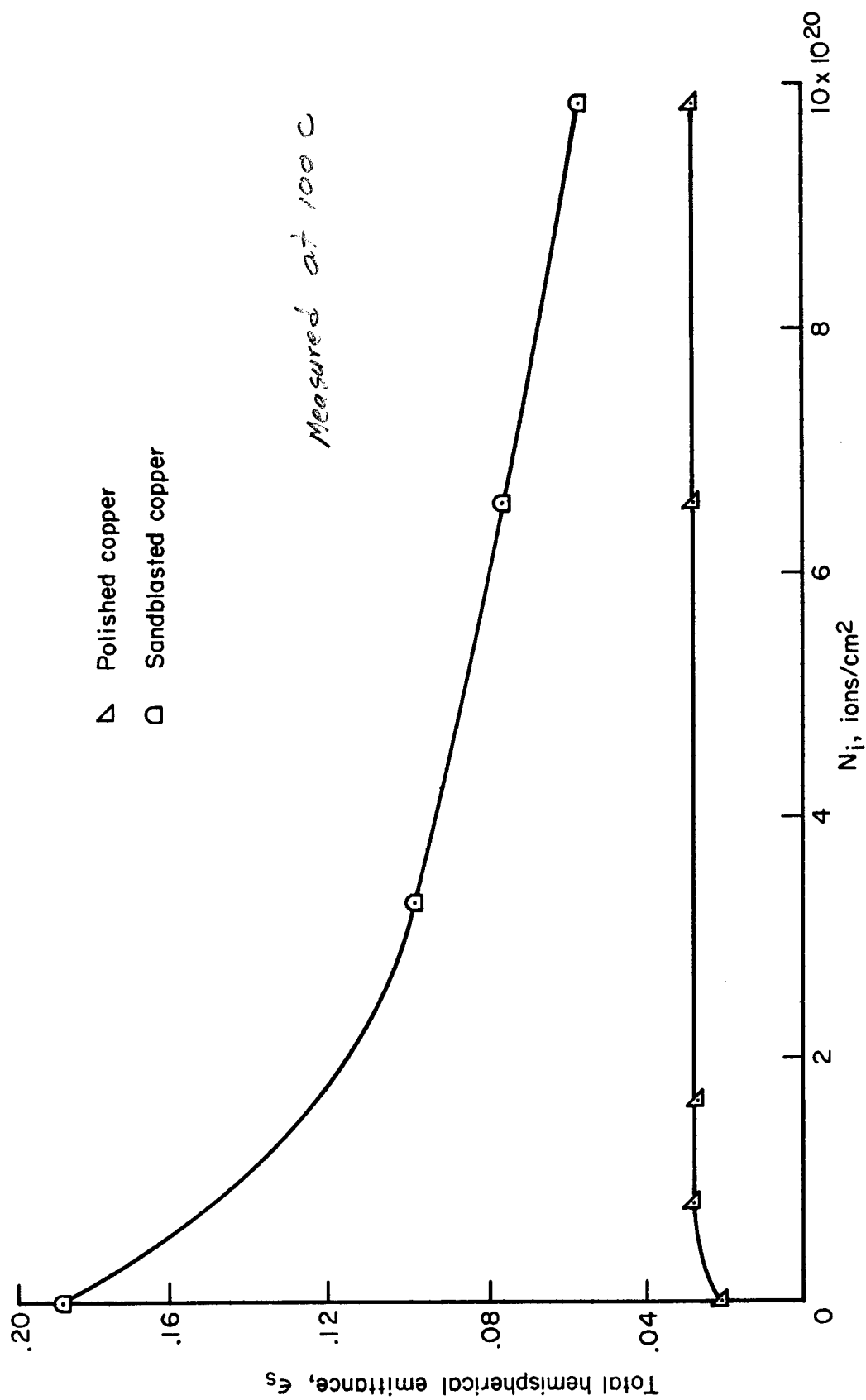
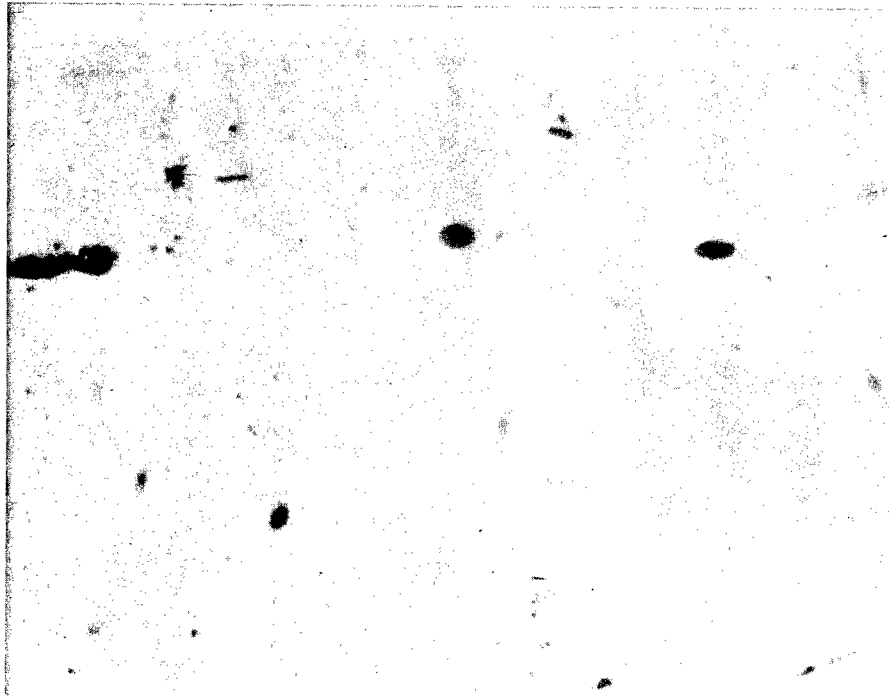


Figure 11.- Effect of hydrogen ion bombardment on total hemispherical emittance of pure copper.



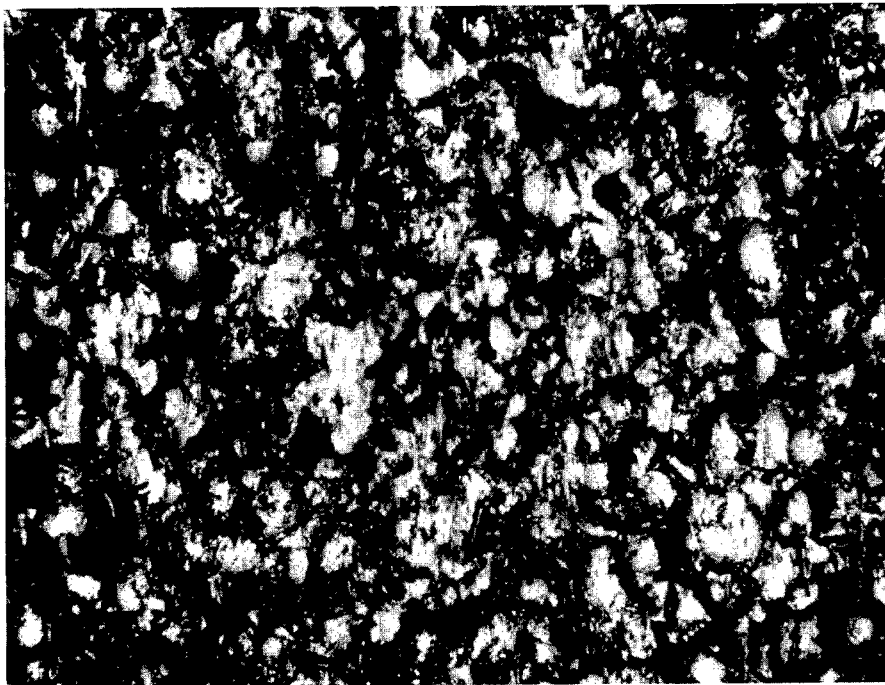
Before ion bombardment.



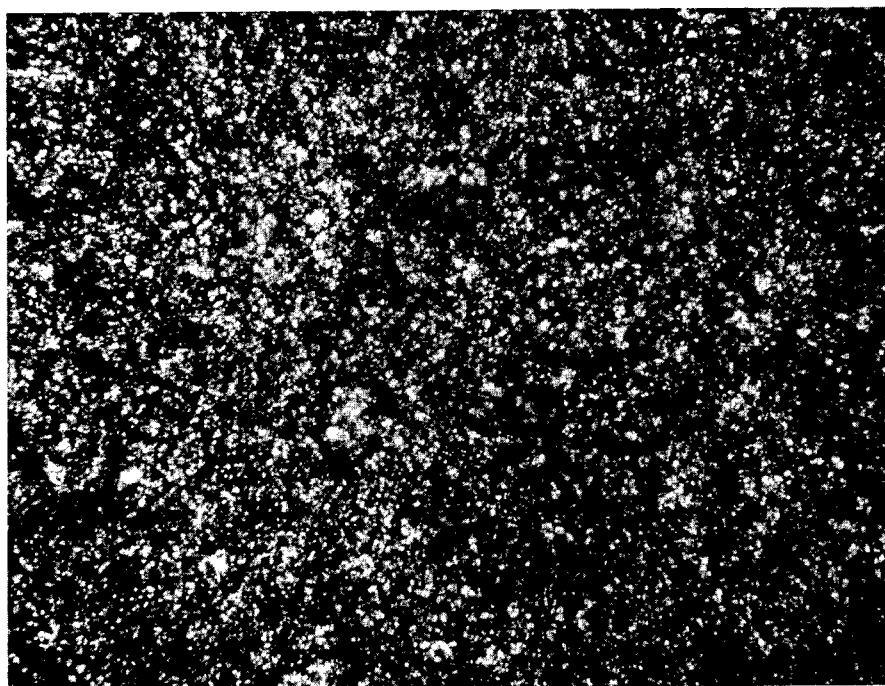
After bombardment with approximately  $10^{21}$  ions/cm<sup>2</sup>.

(a) Initially polished, 500 X.

Figure 12.- Photomicrographs of pure copper specimens before and after sputtering.



Before ion bombardment.



After bombardment with approximately  $10^{21}$  ions/cm<sup>2</sup>.

(b) Initially sandblasted, 250 X.

Figure 12.- Concluded.

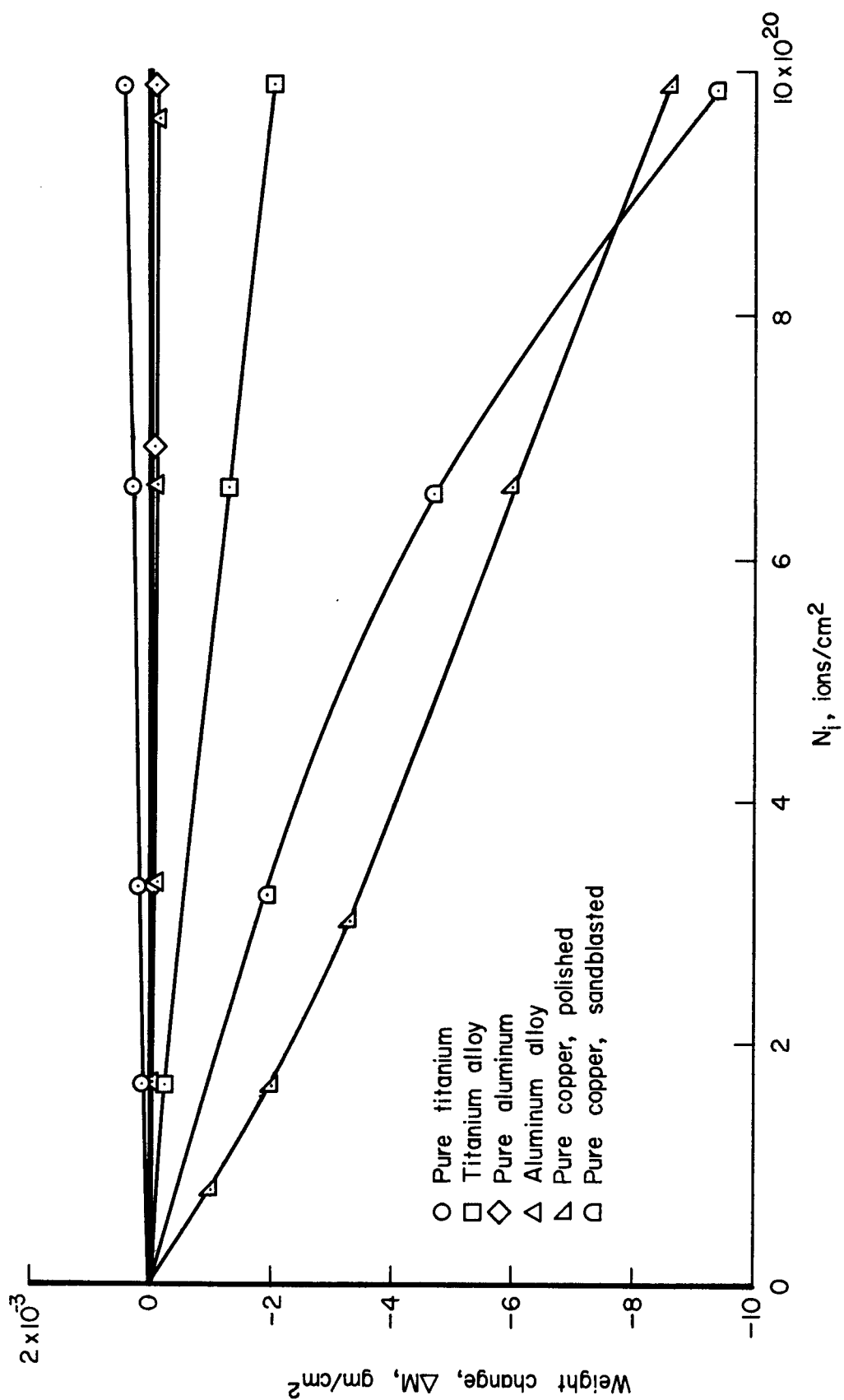


Figure 13.- Weight change of the six test specimens as a function of number of bombarding ions.

<p>NASA TN D-1646 National Aeronautics and Space Administration. EFFECTS OF SPUTTERING WITH HYDROGEN IONS ON TOTAL HEMISPHERICAL EMITTANCE OF SEVERAL METALLIC SURFACES. Donald L. Anderson and George J. Nothwang. January 1963. 36p. OTS price, \$1.00. (NASA TECHNICAL NOTE D-1646)</p> <p>The test specimens were cylindrical and were constructed from pure titanium, a titanium alloy containing 6-percent aluminum and 4-percent vanadium, pure aluminum, 2024 aluminum alloy, and pure copper. The energy level of the incident ions was 1000 electron volts. The test specimens were subjected to ion bombardment by immersion in a hydrogen plasma. The total hemispherical emittance of each specimen was measured in a cold-wall, vacuum-type calorimeter. Photomicrographs of all test specimens before and after bombardment with 1021 ions/cm<sup>2</sup> are presented and discussed. In addition, the changes in weight resulting from ion bombardment of all specimens are presented.</p>	<p>I. Anderson, Donald L. II. Nothwang, George J. III. NASA TN D-1646</p>	<p>NASA TN D-1646 National Aeronautics and Space Administration. EFFECTS OF SPUTTERING WITH HYDROGEN IONS ON TOTAL HEMISPHERICAL EMITTANCE OF SEVERAL METALLIC SURFACES. Donald L. Anderson and George J. Nothwang. January 1963. 36p. OTS price, \$1.00. (NASA TECHNICAL NOTE D-1646)</p> <p>The test specimens were cylindrical and were constructed from pure titanium, a titanium alloy containing 6-percent aluminum and 4-percent vanadium, pure aluminum, 2024 aluminum alloy, and pure copper. The energy level of the incident ions was 1000 electron volts. The test specimens were subjected to ion bombardment by immersion in a hydrogen plasma. The total hemispherical emittance of each specimen was measured in a cold-wall, vacuum-type calorimeter. Photomicrographs of all test specimens before and after bombardment with 1021 ions/cm<sup>2</sup> are presented and discussed. In addition, the changes in weight resulting from ion bombardment of all specimens are presented.</p>	<p>NASA</p>
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